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(54) Process for the preparation of concentrated extracts of coffee, tea or substitutes therefor

(57) A process for the preparation of a concentrated extract of coffee, tea or substitutes therefor, which comprises the steps of
(a) subjecting an extract to an ultra-filtration treatment using a membrane having a molecular weight exclusion point in the range 500 to 5000 Daltons, so as to obtain a retained fraction containing essentially soluble solids and an aromatic permeated fraction;
(b) subjecting ultra-filtration permeated fraction from step (a) to a reverse osmosis treatment using a membrane which is capable of retaining at least 99% of NaCl; and
(c) mixing the retained fraction from the reverse osmosis of step (b) and the retained fraction from the ultra-filtration of step (a) to give the desired concentrated extract. The extract may be subjected to steam distillation before step (a) and the aromas so obtained added to the final concentrated extract.

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SPECIFICATION

Process for the preparation of concentrated extracts of coffee, tea or substitutes therefor

The present invention relates to a process for the preparation of a concentrated extract of coffee, tea or substitutes therefor. The concentrated extract can subsequently be converted into powder by conventional methods.

In the preparation of, for example, instant coffee powders, one of the difficult stages is the concentration of the final extract, said concentration having to be effected with special care to avoid damage to the aroma. In general, moreover, since the concentration is effected by evaporation, the aromas are removed from the extract to be concentrated and subsequently reintroduced into the extract after concentration. However, heat treatment is unavoidable in such a system and losses or changes of aroma can thereby arise.

Attempts have hitherto been made to treat coffee extracts by ultra-filtration with moderate success. In particular, the use of membranes conventional in the art, which have molecular weight exclusion points of the order of 10,000 Daltons and above, has given fractions of little interest and which are difficult to treat by physical means such as reverse osmosis.

The present invention, which employs similar physical processes to those mentioned above, provides a solution of these difficulties.

Thus, according to the present invention there is provided a process for the preparation of a concentrated extract of coffee, tea or substitutes therefor, for example chicory and various herbal infusions and decoctions, which comprises the steps of
(a) subjecting an extract of coffee tea or substitutes therefor to an ultra-filtration (hereafter referred to as UF) treatment using a membrane having a molecular weight exclusion point in the range 500 to 5000 Daltons, so as to obtain a retained fraction containing essentially soluble solids and an aromatic permeate fraction;
(b) subjecting the ultra-filtration permeate fraction from step (a) to a reverse osmosis (hereafter referred to as RO) treatment using a membrane which is capable of retaining at least 99% of NaCl; and
(c) mixing at least part of the retained fraction from the reverse osmosis step (b) and at least part of the retained fraction from the ultra-filtration step (a) to give the desired concentrated extract.

If necessary and/or desired, the starting extract is clarified before being subjected to UF, for example by filtration or by centrifuging.

In an advantageous embodiment of the process, the retained fraction from the reverse osmosis and the ultra-filtration retained fraction are mixed after the latter has been concentrated.

In another embodiment, the retained fraction is diluted while UF is being effected, this operation being referred to as "diafiltration" (DF).

Of course UF and DF operations can be combined either in series or in parallel.

It could be argued that an operation using a membrane having an exclusion point from 500 to 5000 could equally be regarded as "ultra-filtration" or as "reverse osmosis". In fact, the exclusion conditions are such that the process takes place at the limits of the range common to both operations, UF and RO both involving a segregation and a fractionation at the molecular level.

We have however deliberately chosen the term "UF", both for clarity of description and because stage (a) of the process according to the invention involves, above all, retaining a maximum of coffee solids, while allowing the aromas to pass. In contrast, the purpose of stage (b) is to collect the aromas in the retained fraction from the RO, ideally water alone passing through the membrane.

In the process of the invention, the conditions for effecting UF and RO are those conventionally used and, for example, with regard to temperature, the conditions to be selected are governed essentially by the desire to handle the extract as gently as possible. Thus, a temperature of from 2 to 25°C, preferably from 5 to 25 °C, may be used. The pressure in the UF treatment is generally of the order of 4 to 20, preferably 8 atmospheres, and in the RO treatment of 30 to 80 atm. The membranes used may be those conventional in the art, for example membranes of polysulphones or cellulose acetate for UF and membranes of poly-(ether)-amides or polyamides for RO, provided that the exclusion point of the UF membrane is between 500 and 5000, and that the RO membrane is capable of retaining at least 99% NaCl (e.g. sea-water desalination membranes).

In order to enable as much as possible of the aromas to pass through the membrane in stage (a), the retained fraction can, if desired, be diluted with water during the UF treatment. This enables the ratio of solids to aromas in the retained fraction to be increased.

For example, the UF treatment enables ratios of solids to aromas in the retained fraction ranging up to 3.5 to be obtained. The leakage of solids to the permeate fraction still remains minimal; their presence in small quantity is, moreover, advantageous as an aroma carrier during the subsequent RO operation. Furthermore, a ratio of concentration of aromas of the order of 30 to 40 times may be achieved in the retained fraction from the RO treatment; the leakage of aromas to the permeate fraction from the RO treatment is generally insignificant. Advantageously, the UF treatment is continued until a concentration factor of form 2 to 5, e.g. of the order of 3, is obtained, and with the RO concentration factors of between 5 and 10 are aimed at.

The desired concentrated coffee extract is obtained by combining the retained fraction from the RO treatment, obtained as described above, with the retained fraction from the UF treatment. Advantageously, the retained fraction from UF is concentrated by a conventional method before it is added to the retained fraction from RO.

It goes without saying that a partial recombination may be effected by mixing only a part of the retained fraction from RO with the retained fraction from UF or vice versa, and in this way, extracts which are more or less aromatic or of variable aromatic quality may be obtained, in comparison with extracts cor-

responding to those obtained with the total recombination.

The process according to the invention which utilises UF and RO can likewise be supplemented by conventional operations in coffee technology, such as evaporation or steam distillation of aromas.

Thus, in another embodiment of the process according to the invention a partial steam distillation of the starting extract may be effected before UF, the aromas thus collected being added to the final concentrated extract. In yet another embodiment traces of aromas which may have been retained with the solids in the retained fraction from UF are removed by steam distillation and are reintroduced into the final concentrated extract (or into the concentrated retained fraction from UF, if said fraction has undergone such a treatment).

The following Examples illustrate the process according to the invention. In the Examples all percentages are expressed as percentages by weight.

Example 1

An industrial coffee extract obtained by a conventional process is treated by ultra-filtration in a DDS 35.9.3 module equipped with GR 8 P polysulphone membranes. The membranes have a mean exclusions point of 2500 Daltons. The ultra-filtration is effected at a pressure of $7x10^5$ $N.m^{-2}$ (7 bars) at 20° C. UF is continued until a concentration factor of 2 is obtained. The permeate obtained is then treated by reverse osmosis in a module equipped with PA 300 TFC poly-(ether)-amide membranes which are capable of retaining at least 99.4% of NaCl. The reverse osmosis operation is effected at $60x10^5$ $N.m^{-2}$ (60 bars) at 10°C. RO is continued until a concentration factor of 10 is obtained. Under these conditions it is noted that 99% of the solids are found in the retained fraction from ultra-filtration and it is estimated that more than 90% of the aromas, also, are found in the retained fraction from reverse osmosis. The whole of the retained fraction from reverse osmosis and the retained fraction from ultra-filtration which are obtained above are then mixed. The mixture is converted into instant coffee powder according to conventional method.

The powder reconstituted in water gives a coffee of excellent quality whose aroma, in particular, is judged to be "very true".

Example 2

The procedure described in Example 1 is repeated, but the ultra-filtration is continuous until a concentration factor of 4 is obtained. The results obtained are very comparable, the mixtures of the retained fractions from ultra-filtration and from reverse osmosis have a slightly lower concentration and thus make it necessary to evaporate a little more water to produce the instant coffee powder.

Example 3

The procedure described in Example 1 is repeated, but, onc the concentration factor of 2 is obtained in the ultra-filtration step, the operation is continued by diluting the ultra-filtration retained fraction (diafiltration). For diafiltration a quantity of water equal to half the quantity of coffee extract used in the operation is employed. The reverse osmosis operation proceeds as described in the foregoing Examples.

By drying the mixture of the retained fractions from reverse osmosis and from ultra-filtration (diafiltration) a powder is obtained which, after reconstitution, gives a coffee of excellent quality.

The losses of aroma and of coffee solids may be considered as negligible.

Example 4

The procedure described in Example 1 is repeated, with the following two differences: on the one hand, the ultra-filtration is effected at a pressure of $15x10^5$ $N.m^{-2}$ (15 bars) and, on the other hand, the ultra-filtration retained fraction is subjected to a steam distillation operation at 100°C for one minute, the aromas thus collected being added to the final mixture before drying.

Example 5

The procedure described in Example 2 is repeated, but the retained fraction obtained at the end of the ultrafiltration operation which has a content of dry substances of 12.2% is concentrated thermally until a content of dry substances of 56% is obtained. This concentrated retained fraction is added in this form to the retained fraction from reverse osmosis to constitute the final mixture which, after drying, gives instant coffee powder.

Example 6

An industrial coffee extract is ultra-filtered in a DDS module equipped with cellulose laboratory membranes having an exclusion point of 500 Daltons. The pressure applied is $30x10^5$ $N.m^{-2}$ (30 bars) and the temperature is 20°C. The operation is stopped when a concentration factor of 2 is obtained.

The ultra-filtration permeate is treated by reverse osmosis in a module equipped with PA 300 TFC poly-(ether)-amide membranes, as indicated above The operation is effected at a pressure of $60x10^5$ $N.m.^{-2}$ (60 bars) and a temperature of 10°C and is limited to a concentration factor of 4.

The retained fractions from reverse osmosis and from ultra-filtration are combined to give the mixture from which instant coffee powder is prepared.

Example 7

The ultra-filtration operation described in Example 1 is repeated, starting from an industrial coffee extract which has previously been concentrated thermally to a concentration factor of 3. Subsequently, the ultra-filtration permeate is subjected to a reverse osmosis operation, as described in the foregoing Example 6.

A combination of the two retained fractions from reverse osmosis and from ultra-filtration gives the mixture from which instant coffee powder is obtained.

COMPARATIVE EXAMPLE

An industrial coffee extract is treated by ultrafiltration in a module equipped with Romicon PM 50 membranes of the polysulphonic type having an exclusion point of 50,000 Daltons. The operation is effected at $1.5x10^5$ $N.m^{-2}$ (1.5 bars) at 20°C until a concentration factor of 4 is obtained. The distribution of solids between the retained fraction and the permeated fraction is catastrophic since at least 30% of the solids can be found in the permeated fraction. Furthermore, the aromas are distributed approximately equally between the retained fraction and the

permeate fraction.

The ultra-filtration permeate is then treated by reverse osmosis under the conditions described in the other Examples. The pressure is 60×10^5 $N.m^{-2}$ (60 bars) and the temperature is 10°C and the operation is continued until a concentration factor of 4 is obtained.

A combination of the retained fractions from reverse osmosis and from ultra-filtration gives a mixture which can be converted directly into an instant coffee powder, but it is noted that this powder has a reduced aromatic value. The considerable loss of aroma during ultra-filtration is manifestly the reason for this. Moreover, the loss of solids which is also considerable makes the operation of little potential interest on an industrial scale.

CLAIMS

1. A process for the preparation of a concentrated extract of coffee, tea or substitutes therefor which comprises the steps of
(a) subjecting an extract of coffee, tea or substitutes therefor to an ultra-filtration treatment using a membrane having a molecular weight exclusion point in the range 500 to 5000 Daltons, so as to obtain a retained fraction containing essentially soluble solids and an aromatic permeate fraction;
(b) subjecting the ultra-filtration permeate from step (a) to a reverse osmosis treatment using a membrane which is capable of retaining at least 99% of NaCl; and
(c) mixing at least part of the retained fraction from the reverse osmosis step (b) and at least part of the retained fraction from the ultra-filtration step (a) to give the desired concentrated extract.

2. A process according to claim 1 wherein the retained fraction from the reverse osmosis step (b) and the retained fraction from the ultra-filtration step (a) are mixed after the latter has been concentrated.

3. A process according to either of claims 1 and 2, wherein the retained fraction in step (a) is diluted during ultra-filtration.

4. A process according to any one of the preceding claims wherein the ultra-filtration and reverse osmosis treatments are effected at a temperature of from 2 to 25°C.

5. A process according to any one of the preceding claims wherein the ultra-filtration treatment is effected at a pressure of from 4 to 20 atmospheres.

6. A process according to any one of the preceding wherein the reverse osmosis treatment is effected at a pressure of between 30 and 80 atmospheres.

7. A process according to any one of the preceding claims wherein the ultra-filtration treatment is effected so as to obtain a concentration factor of from 2 to 5.

8. A process according to any one of the preceding claims wherein the reverse osmosis treatment is effected so as to obtain a concentration factor of from 5 to 10.

9. A process according to any one of the preceding claims wherein the mixing between the retained fraction from reverse osmosis of step (b) and the retained fraction from ultra-filtration of step (a) is only partial.

10. A process according to any one of the preceding wherein steam distillation of the starting extract is effected before ultra-filtration, the aromas thus obtained being added to the final concentrated extract.

11. A process according to any one of the preceding claims wherein steam distillation of the retained fraction from ultra-filtration is effected, the aromas thus obtained being added to the final concentrated extract.

12. A process for the preparation of a concentrated extract of coffee, tea or substitutes therefor substantially as herein described in any one of Examples 1 to 7.

13. A concentrated extract of coffee, tea or substitutes therefor which is obtained by a process according to any one of the preceding claims.

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